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## Persistence and Phytotoxicity of Pendimethalin Herbicide in Soils of the Aceh Province, Indonesia

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### Abstract

Field studies were undertaken to evaluate the residual phytotoxicity and persistence of pendimethalin herbicide. The doses of pendimethalin 0.4, 0.8 and 1.2  $\mu\text{g} \cdot \text{g}^{-1}$  dry weight of soil were applied to pots containing soils from site of Bireun, Lhoksukon, and Bener Meriah (Aceh Province, Indonesia). Residual phytotoxicity and chemical persistence at 30, 60 and 90 days after treatment (DAT) were studied by water melon bioassay and gas chromatography. Residual phytotoxicity values obtained at all time and up to a maximum of 120 DAT followed the order Bener Meriah>Lhoksukon>Bireun. A highly negative ( $P<.01$ ) correlation between the percentage of pendimethalin mass chemically recovered and its residual phytotoxicity at every comparable sampling time, for doses ranging from 0.8  $\mu\text{g} \cdot \text{g}^{-1}$  to 1.2  $\mu\text{g} \cdot \text{g}^{-1}$  dry soil weight was obtained. Residual phytotoxicity and chemical persistence in relation to physicochemical properties of soils.

**Keywords:** persistence; residual; phytotoxicity; pendimethalin; soil.

### 1. Introduction

Pendimethalin is dinitroaniline herbicide that usually used for selective control of most annual grasses and many annual broad-leaved weeds in several crops in Indonesia. Pendimethalin was adsorbed by the roots and leaves, and it inhibits cell division and cell elongation [1]. In Indonesia it was applied in soybean (*Glycine max* L. Merrill), Maize, and onion (*Allium cepa*) crops.

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The preemergence application of pendimethalin to control weed in soybean was recommended in Indonesia. In Indonesia the chemical weed control as the most efficient methods used in weed control, occasionally their uses are not feasible as a result of unsuitable applying time, such as sprayed in the late stages of the weed's life [2], but environmental issues are pressing farmers to reduce costs and pesticide use in Indonesia and other parts of the world [3]. Persistence of pendimethalin in soil was influenced by cultivation practices, soil temperature, moisture conditions and soil type. Photodecomposition can occur, especially during the first days of exposure on the soil surface [4]. Field studies indicate that the herbicide is persistent and not leaching [5]. Pendimethalin residues in soil have been studied in world [6,4]. Information concerning the persistence of residues of Pendimethalin herbicide in soil that cultivated by soybean was quite limited [7]. Based on literature review, however there was no research about pendimethalin herbicide that already carried out in Indonesia. Most of the method used to determine the presence of pendimethalin in soil are complicated dan expensive. On other hand, the detection of traces of herbicide does not represent a direct measure of phytotoxic effect. A biotest using plant spesies that sensitive to the herbicide as control may statisfy both requirements. The objective of this study was to evaluate the persistence of pendimethalin in three representative soils of the Aceh province, Indonesia, using water melon bioassay and chemichal extraction followed by Gas Chromatographi (GC).

## 2. Materials and Methods

### 2.1 Bioassay

Soil samples were collected from 0-20 cm depth at Bireun, Lhoksukon and Bener Meriah, Aceh Province, Indonesia. All sample was previously homoganized, air dried and passed through a 1-2mm sive before use. The Properties of these soils are given in Table 1.

**Table 1:** Properties of Soil in the Studied

Site	Texture	Sand	Silt (%)	Clay (%)	OM (%)	pH	CEC
		(% )					
Bireun	Loam	35.2	28.0	26.8	3.2	5.4	30.1
Lhoksukon	Clay loam	25.1	45.1	29.8	2.8	5.2	26.2
B.Meriah	Clay loam	39.1	30.1	30.8	5,8	6,2	36,3

OM : Organic matter, CEC : cation exchange capacity (meq 100 g<sup>-1</sup> soil)

Soil samples were put in pots, each one constituting an experimental sample. An Emulsifiable concentration formulation of pendimethalin at 33% active ingredient (ai) was used. Herbicide was applied to soil in pots to achieve concentration of 0.4, 0.8, and 1,2 µg. g<sup>-1</sup>. These are equivalent to the dosage of 1.0, 2,0 and 4 kg a.i ha<sup>-1</sup>

in the field. Each sample was thoroughly mixed to allow a homogeneous distribution. Non sprayed soil samples were used as controls. Completely randomized design with factorial arrangement was used in this research. Each treatment was replicated 5 times. The activity of herbicide was evaluated using a water melon bioassay. After pendimethalin was added to the soil, 5 watermelon seed were planted per pot. After seedling establishment, plant number was thinned to 3 per pot. The seed bed was moistened daily with distilled water to reach 75% field capacity and fertilized with a superphosphate (36%  $P_2O_5$ ) water solution, to achieve a final concentration of 96  $\mu\text{g g}^{-1}$ . The plants were grown in a greenhouse under controlled condition. Shoot growth impairment (SGI) due to herbicide activity was defined as the percent of loss in shoot dry weight, when plant grown on soils that treated with pendimethalin were compared with control (soil that not treated with herbicide). The analysis of variance was done for each soil and treatment mean were compared by LSD. The concentration of pendimethalin required in soils to reduce shoot dry weights of water melon 50% ( $GR_{50}$ ) were estimated. The number of DAT required to obtain **SGI** values that lower than 20%, was arbitrarily adopted as a way to compare practical residual phytotoxities among soils and calculated in similar way as for  $GR_{50}$ .

## **2.2 GC Analysis**

The parallel and similar assay to the watermelon bioassay with the same soils and treatment as described earlier, was also carried out to study and compare the obtained result. Soil samples were collected at 0, 30, 60, 90 day after application of pendimethalin. At each date of sampling a composite sample of 200 g soils was taken from each treatment. Soil extraction of pendimethalin residues was performed by ethyl acetate. The 20 g soil samples were extracted with 100 mL of ethyl acetate by shaking on a wrist action shaker for 1 hours, and then the extract was centrifuged. The soil was reextracted and centrifuged as above. The supernatants were filtered through Whatman no. 1 filter paper, and the filter was washed with additional ethyl acetate. The filtrate was evaporated to dryness, under vacuum, using a rotary evaporator (40°C). The residue was dissolved with a suitable volume of ethyl acetate, and the aliquot was analyzed by gas chromatography. A Hewlett-Packard model 6890 gas chromatograph, equipped with a Ni-63 electron capture detector and with a 30 m x 0.32 mm i.d. fused silica capillary column coated with 0.25  $\mu\text{m}$  of 5% phenyl methyl siloxane (HP-5), was used for the determination of the herbicide. The following operating conditions were used: carrier gas (helium) flow at 2.8  $\text{mL min}^{-1}$ ; makeup gas (nitrogen) flow at 30  $\text{mL min}^{-1}$ ; injector temperature at 270 °C; detector temperature at 300 °C. All samples (2  $\mu\text{L}$ ) were injected with the injector in the pulsed splitless mode (40 psi for 1.0 min) and after the analysis was taken at constant flow. The oven temperature program used was as follows: 60 °C (1 min) to 150 °C at 10 °C  $\text{min}^{-1}$ , to 180 °C (0 min) at 3 °C  $\text{min}^{-1}$ , to 205 °C (2 min) at 10 °C  $\text{min}^{-1}$ , to 260 °C (8 min) at 30 °C  $\text{min}^{-1}$ . Under these conditions, the retention time for pendimethalin was 22.2 min. The temperature program, used for separation and determination of pendimethalin, was found to be efficient for separating and determining other herbicides as well.

## **3. Results and Discussions**

### **3.1 Residual Phytotoxicity**

Table 2 shown the SGI evolution in shoots from four consecutive crops of Water melon in soils from Aceh

Province, Indonesia that previously treated with single doses of pendimethalin applied at three different concentration. The toxicity of pendimethalin expressed as the amount in the soil which reduced the growth of watermelon by 50% ( $GR_{50}$ ), could be calculated by using a data at Table 2 and found the values of 0.4, 0.8, and 1.2  $\mu\text{g. g}^{-1}\text{DSW}$  in soils from Bireun, Lhoksukon and Bener Meriah. The phytotoxicity of pendimethalin expressed in term of  $GR_{50}$  followed the order: Bener Maeriah > Lhoksukon > Bireun. Even though our result are insufficient to asses a relation among soil parameters and  $GR_{50}$  values, Bener Meriah soil had both the high organic matters (OM) and cation exchange capacity (CEC), and the highest pH of all the soils under study (Table 1). All the the parameters have been shown to enhance pendimethalin bioavailability in soils.

**Table 2:** Shoot growth impairment (SGI) at days after treatment (DAT) in water melon crops obtained from soil treated pendimethalin

Soil site	Dose $\mu\text{g. g}^{-1}\text{DSW}$	Day after treatment			
		30	60	90	120
		(%)	(%)	(%)	(%)
Bireun	0	0 <sup>a</sup>	0 <sup>a</sup>	0 <sup>a</sup>	0 <sup>a</sup>
	0.4	45.1 <sup>b</sup>	16.7 <sup>bc</sup>	15.3 <sup>b</sup>	3.1 <sup>ab</sup>
	0.8	80.2 <sup>c</sup>	29.1 <sup>c</sup>	17.7 <sup>b</sup>	5.3 <sup>ab</sup>
	1.2	89.6 <sup>ef</sup>	59.1 <sup>e</sup>	26.0 <sup>d</sup>	5.5 <sup>ab</sup>
Lhoksukon	0	0 <sup>a</sup>	0	0 <sup>a</sup>	0 <sup>a</sup>
	0.4	44.1 <sup>b</sup>	17.1 <sup>bc</sup>	15.8 <sup>b</sup>	4.1 <sup>a</sup>
	0.8	83.2 <sup>c</sup>	29.8 <sup>c</sup>	18.9 <sup>c</sup>	6.3 <sup>b</sup>
	1.2	84.6 <sup>e</sup>	59.7 <sup>e</sup>	28.8 <sup>d</sup>	7.5 <sup>bc</sup>
Bener Meriah	0	0	0 <sup>a</sup>	0 <sup>a</sup>	0
	0.4	54.1 <sup>d</sup>	26.7 <sup>c</sup>	16.7 <sup>b</sup>	5.1 <sup>ab</sup>
	0.8	88.2 <sup>ef</sup>	39.1 <sup>d</sup>	29.1 <sup>d</sup>	7.3 <sup>bc</sup>
	1.2	94.6 <sup>f</sup>	66.1 <sup>f</sup>	59.1 <sup>e</sup>	18.5 <sup>d</sup>

<sup>a-f</sup>Mean followed by the same letter in columns are not significantly different ( $P < 0.05$ ) according to LSD Test (Percent of control)

The interaction effect between soil and dose was found for the four periods of evaluation. Shoot growth impairment at 30 and 60 DAT were significantly higher than controls for all the pendimethalin doses that applied to the soils under study. However, after 120 day of herbicide treatment, shoot growth impairments (SGI) were not significantly different from the controls in Bireun soils at 0.4, 0.8 and 1.2  $\mu\text{g. g}^{-1}$  DSW doses (Table 2). This trend was also evident after 120 day for Lhoksukon and Bener Meriah soils, at each herbicide concentration. Bener Meriah soil on the other hand showed some herbicidal effects on water melon shoot growth even after 120 day of pendimethalin treatment at 0.4, 0.8, and 1.2  $\mu\text{g. g}^{-1}$  DSW doses (Table 2).

SGI obtained 30 day after pendimethalin treatment was not significantly different ( $P < .05$ ) among soils when the herbicide was applied at the dose of 0.8 and 1.2  $\mu\text{g. g}^{-1}$  DSW. According to SGIs, the residual phytotoxicities followed the order Bener Meriah > Lhoksukon > Bireun at 0.4  $\mu\text{g. g}^{-1}$  DSW pendimethalin doses. On the other hand Table 2 showed there were significantly higher in Bener Meriah soil than in the other soil (Bireun and Lhoksukon). The practical purpose of soil which causes less than 20% reduction in growth in pendimethalin-sensitive plants could be considered adequate for sowing commercial crops. Table 2 presented could be useful to calculate the waiting period prior to crop rotation. The herbicide persistence obtained here is in accordance with that reported for temperate soils where a field rate of 1.5 kg ai.  $\text{ha}^{-1}$  of pendimethalin is expected to give a full season's control of between 3 -12 months. In contrast the herbicidal persistence of pendimethalin in tropical soil lasted no longer than 70 days (4).

The toxicity and persistence pendimethalin were related to the organic matter content of soil due to greater retention of the vapour phase of the herbicide [8]. Reference [9] also revealed that pendimethalin persistence was dependent on organic matter rather than clay content. However, the clay fraction contributed more to the reduction of phytotoxicity than the organic matter fraction [10]. In contrast to this, the incorporation of colloidal fraction of leonardite or activated charcoal in mineral soils of low organic matter resulted in increased toxicity of surface applied pendimethalin. When pendimethalin was incorporated 2.5 cm in soil, no influence was observed from increased organic matter [8]. Reference (11) revealed that phytotoxicities of pendimethalin decreased with increased organic matter. At the end of the persistence assay (120 DAS), the herbicides residues in soil. These values were the same as reported by [12]. This may be due to dinitroanilines being degraded by chemical reactions, biological processes [13] and volatility [14].

### **3.2 Chemical Persistence**

Table 3 shows the percentage of pendimethalin mass recovered versus time, in Bireun, Lhoksukon, and Bener Meriah upper layer soils (0 – 20 cm) and the different application rates. Mass recovery rates of pendimethalin 120 DAT varied among soils and doses, and ranged between 17 – 8.5%, 19.5 – 7.8% and 16.4 – 12.5% in Bireun, Lhoksukon, and Bener Meriah soils (Table 3). These result were in accordance with Walker (1978) who obtained residues 5 months after application generally less than 20% of the amount initially present. Table 3 shows a higher half-life for pendimethalin in Lhoksukon than the other soils when 0.58  $\mu\text{g. g}^{-1}$  DSW pendimethalin doses was used. At 0.8  $\mu\text{g. g}^{-1}$  DSW doses of pendimethalin herbicide half life is about the same for all soils, and at 1.2  $\mu\text{g. g}^{-1}$  DSW pendimethalin doses herbicide half life could be ordered as follow Bireun > Lhoksukon > Bener Meriah (Table 3).

**Table 3:** Pendimethalin mass recovered (per cent of initial concentration) and calculated half-life for different soils and application doses.

Soil site	Dose $\mu\text{g. g}^{-1}\text{DSW}$	Day after treatment			Half-life (days)
		30 (%)	60 (%)	90 (%)	
Bireun	0.4	65.1	37.3	15.1	74
	0.8	50.2	32.2	11.3	45
	1.2	45.6	25.6	8.50	38
Lhoksukon	0.4	88.1	46.2	18.8	99
	0.8	50.1	25.7	18.9	43
	1.2	31.2	9.7	7.8	31
Bener Meriah	0.4	72.9	16.7	14.7	65
	0.8	51.3	35.1	12.1	47
	1.2	57.3	31.6	15.1	58

The highest residual phytotoxicity was obtained in Bener Meriah soil, independently of the initial pendimethalin dose and sampling time (Table 2). Chemical persistence and phytotoxicity of pendimethalin for different soils and doses could be compared at 60, 90, and 120 DAT (Table 4).

**Table 4:** Correlation coefecient (r) between pendimethalin residual phytotoxicity (as SGI) and chemical persistence (as percent of mass recovery) for different soils and doses.

Soil	Dose ( $\mu\text{g. g}^{-1}\text{DSW}$ )		
	0.4	0.8	1.2
Bireun	- 0.71**	- 0.85**	- 0.94**
Lhoksukon	- 0.68**	- 0.96**	- 0.77**
Bener Meriah	- 0.78**	- 0.91**	- 0.42*

\* and \*\* statistically significant at 5 or 1%, respectively

Table 4 shows a high correlation between the percentage of pendimethalin mass chemically recovered and its residual phytotoxicity (SGI) in all soils and at every comparable sampling time, for a dose of  $0.8 \mu\text{g. g}^{-1}$ . This dose in pots is equivalent to the field rate ( $1.5 \text{ kg ai. ha}^{-1}$ ). The obtained through a simpler phytotoxicity test, is

comparable to the one obtained through the more complicated and expensive GC analysis when pendimethalin is used at the recommended dose in Bener Meriah and Lhoksukon soils. Considering the result in Table 2, a minimum waiting period of 75, 90, and 120 day should be considered prior to sowing pendimethalin-sensitive crops in Lhoksukon, Bireun and Bener Meriah after recommended dose of pendimethalin is applied to the soils. This result, however obtained through a simpler phytotoxicity test, is comparable to the one obtained through the more complicated and expensive GC analysis when pendimethalin used at the recommended dose in Bener Meriah, Bireun Lhoksukon soil (Table 4).

Persistence of pendimethalin can be influenced by many factors including soil temperature, soil moisture, type of soil, cultivation practices [15] and soil organic matter [16]. Reference [17] observed that pendimethalin was more persistent when incorporated in the soil than applied to the soil surface. Reference [14] revealed that pendimethalin was not affected by a few days delay in incorporation, however, losses increased if incorporation in the soil was delayed for more than three days [18]. When pendimethalin was applied under no-till conditions, it was found to be most persistence in silty clay and least persistence in sandy loam [19,20].

#### **4. Conclusions**

In this study persistence and phytotoxicity of pendimethalin herbicide in soils was evaluated with assay and chemical in Aceh Province, Indonesia. Persistence herbicide pendimethalin was slightly higher at Bener Meriah than Lhoksukon and Bireun. Application of pendimethalin herbicides do not post any problem with respect to long term herbicide residual contamination in soil.

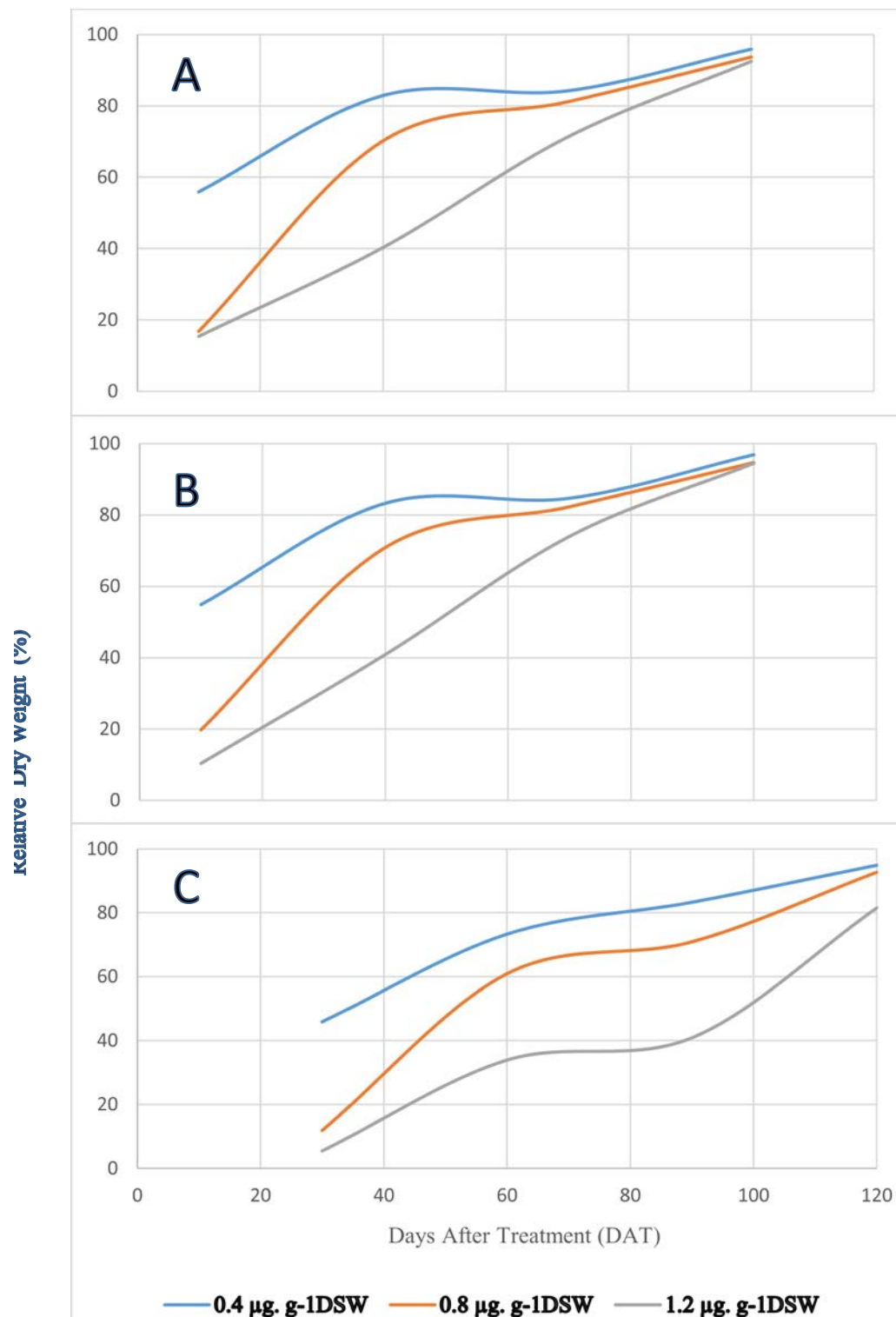
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**Figure 1:** Shoot relative dry weight at Day after treatment obtained consecutive watermelon crops grown in soils from Lhoksukon (A), Bireun (B), and Bener Meriah (A), treated with different doses pendimethalin.